
**Petroleum and natural gas industries —
Completion fluids and materials —**

**Part 5:
Procedures for measuring the long-term
conductivity of proppants**

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*Industries du pétrole et du gaz naturel — Fluides de complétion et
matériaux —*

*Partie 5: Modes opératoires pour mesurer la conductivité à long terme
des agents de soutènement*

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 13503-5 was prepared by Technical Committee ISO/TC 67, *Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries*, Subcommittee SC 3, *Drilling and completion fluids, and well cements*.

ISO 13503 consists of the following parts, under the general title *Petroleum and natural gas industries — Completion fluids and materials*:

- *Part 1: Measurement of viscous properties of completion fluids*
- *Part 2: Measurement of properties of proppants used in hydraulic fracturing and gravel-packing operations*
- *Part 3: Testing of heavy brines*
- *Part 4: Procedure for measuring stimulation and gravelpack fluid leakoff under static conditions*
- *Part 5: Procedures for measuring the long-term conductivity of proppants*

Introduction

This part of ISO 13503 is largely based on API RP 61^[1]. Informative references are also included in the Bibliography, References [2] to [15].

The tests and test apparatus herein have been developed to establish standard procedures and conditions for use in evaluating the long-term conductivity of various hydraulic fracture proppant materials under laboratory conditions. This procedure enables users to compare the conductivity characteristics under the specifically described test conditions. The test results can aid users in comparing proppant materials for use in hydraulic fracturing operations.

The procedures presented in this publication are not intended to inhibit the development of new technology, materials improvements, or improved operational procedures. Qualified engineering analysis and sound judgment is required for their application to fit a specific situation.

This part of ISO 13503 may be used by anyone desiring to do so. Every effort has been made by ISO and API to ensure the accuracy and reliability of the data contained in it. However, ISO and API make no representation, warranty, or guarantee in connection with this part of ISO 13503, and hereby expressly disclaim any liability or responsibility for loss or damage resulting from its use or for the violation of any federal, state, or municipal regulation with which this part of ISO may conflict.

In this part of ISO 13503, where practical, U.S. customary units are included in parentheses for information.

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Petroleum and natural gas industries — Completion fluids and materials —

Part 5: Procedures for measuring the long-term conductivity of proppants

CAUTION — The testing procedures in this part of ISO 13503 are not designed to provide absolute values of proppant conductivity under downhole reservoir conditions. Long-term test data have shown that time, elevated temperatures, fracturing fluid residues, cyclic stress loading, embedment, formation fines and other factors further reduce fracture proppant pack conductivity. Also, this reference test is designed to measure only the frictional energy losses corresponding to laminar flow within a pack. It is recognized that fluid velocity within an actual fracture can be significantly higher than in these laboratory tests, and can be dominated by inertial effects.

1 Scope

This part of ISO 13503 provides standard testing procedures for evaluating proppants used in hydraulic fracturing and gravel-packing operations.

NOTE The “proppants” mentioned henceforth in this part of ISO 13503 refer to sand, ceramic media, resin-coated proppants, gravel packing media, and other materials used for hydraulic fracturing and gravel-packing operations.

The objective of this part of ISO 13503 is to provide consistent methodology for testing performed on hydraulic-fracturing and/or gravel-packing proppants. It is not intended for use in obtaining absolute values of proppant pack conductivities under downhole reservoir conditions.

2 Normative reference

The following referenced document is indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced standard (including any amendments) applies.

ISO 3506-1, *Mechanical properties of corrosion-resistant stainless-steel fasteners — Part 1: Bolts, screws and studs*

3 Terms and definitions

3.1

conductivity

width of the fracture multiplied by the permeability of the proppant pack

3.2

laminar flow

type of streamlined flow for single-phase fluids in which the fluid moves in parallel layers, or laminae, such that the layers flow smoothly over each other with instabilities being dampened by the viscosity

3.3

Ohio sandstone

fine-grained sandstone found in the United States from the Scioto Formation in southern Ohio

3.4

permeability

a measure of the ability of media to transmit fluid through pore spaces

4 Abbreviations

API	American Petroleum Institute
ASTM	American Society for Testing and Materials
RTV	Room temperature vulcanizing
ANSI	American National Standards Institute
PID	Proportional-integral device

5 Procedures for evaluating long-term proppant pack conductivity

5.1 Objective

The objective is to establish a standard test procedure, using a standard apparatus, under standard test conditions to evaluate the long-term conductivity of proppants under laboratory conditions. This procedure is used to evaluate the conductivity of proppants under laboratory conditions but is not intended for use in obtaining absolute values of proppant pack conductivities under downhole reservoir conditions. The effects of fines, formation hardness, resident fluids, time, and/or other factors are beyond the scope of this procedure.

5.2 Discussion

In this part of ISO 13503 procedure, a closure stress is applied across a test unit for $50 \text{ h} \pm 2 \text{ h}$ to allow the proppant sample bed to reach a semi-steady state condition. As the fluid is forced through the proppant bed, the proppant pack width, differential pressure, temperature and flow rates are measured at each stress level. Proppant pack permeability and conductivity are calculated.

Multiple flow rates are used to verify the performance of the transducers, and to determine darcy flow regime at each stress; an average of the data at these flow rates is reported. A minimum pressure drop of 0,01 kPa (0,002 0 psi) is recommended; otherwise, flow rates shall be increased. At stipulated flow rates and temperature conditions, no appreciable non-darcy flow or inertial effects are encountered. After completing the rates at a closure stress level in all cells, the closure stress is increased to a new level; $50 \text{ h} \pm 2 \text{ h}$ is allowed for the proppant bed to reach a semi-steady state condition, and multiple flow rates in all cells are introduced to gather data required to determine proppant pack conductivity at this stress level. The procedure is repeated until all desired closure stresses and flow rates have been evaluated. To achieve accurate conductivity measurements, it is essential that single-phase flow occurs.

Test condition parameters, such as test fluid, temperature, loading, sandstone and time, at each stress shall be reported along with long-term conductivity and permeability data. Other conditions can be used to evaluate different characteristics of proppants and, therefore, can be expected to produce differing results.

6 Reagents and materials

6.1 Test fluid

The test fluid is 2 % by mass potassium chloride (KCl) in a deionized or distilled-water solution filtered to at least 7 µm. The potassium chloride shall be at least 99,0 % by mass pure.

6.2 Sandstone

Ohio sandstone cores should have dimensions of 17,70 cm to 17,78 cm (6,96 in to 7,00 in) in length, 3,71 cm to 3,81 cm (1,46 in to 1,50 in) wide, and a minimum of 0,9 cm (0,35 in) thick. The ends of the sandstone cores shall be rounded to fit into the test unit (see 7.1). Parallel thickness shall be maintained within $\pm 0,008$ cm ($\pm 0,003$ in).

7 Long-term conductivity test apparatus

7.1 Test unit

The test unit shall be a linear flow design with a 64,5 cm² (10 in²) proppant and bed area. Figure C.1 illustrates the details of the test unit and an example of how cells can be stacked. The pistons and test chamber(s) shall be constructed of 316 stainless steel (e.g. ISO 3506-1, Grade A4), Monel¹⁾ or Hastalloy material. Filters for the test unit may be constructed using Monel wire cloth with an opening of 150 µm or equivalent (100 US mesh). Nominal particle retention sizes are greater than 114 µm.

7.2 Hydraulic load frame (standards.iteh.ai)

The hydraulic load frame shall have sufficient capacity to develop 667 kN (150 000 lbf). To ensure uniform stress distribution, the platens shall be parallel to each other. It is recommended that the hydraulic load frame be of a four-post design that minimizes warping that can be transmitted to the test cell. Each post should have a minimum diameter of 6,35 cm (2,5 in).

The hydraulic pressurization source shall be capable of holding any desired closure stress [$\pm 1,0$ % or 345 kPa (50 psi), whichever is greater] for 50 h. The hydraulic load frame shall be capable of loading rate changes of 4 448 N/min (1 000 lbf/min) or 690 kPa/min (100 psi/min) on a 64,5 cm² (10 in²) cell. A calibrated electronic load cell shall be used to calibrate the stress between the hydraulic ram and the opposing platen of the load frame.

7.3 Pack width measurement device(s)

Pack width measurements shall be made at each end of the test unit. A measuring device capable of measuring to 0,002 5 cm (0,001 in) accuracy or better shall be used. Figure C.4 shows an example of width slats allowing for the measurement of pack widths.

7.4 Test fluid drive system

Some constant-flow-rate pumps (e.g. chromatographic pumps) have been found satisfactory for this application. Pulsation dampening can be necessary and can be accomplished by use of a piston, bladder accumulator or other effective means. Pressure fluctuations during differential pressure and flow rate measurements (for conductivity calculations) shall be maintained at less than 1,0 %. Each laboratory shall determine the best technique for pulsation dampening. Large pressure spikes can be indicative of pump problems or trapped gas in the flow system and shall be corrected before recording data.

1) Monel and Hastalloy are examples of suitable products available commercially. This information is given for the convenience of users of this part of ISO 13503 and does not constitute an endorsement by ISO of this product.

7.5 Differential pressure transducers

Differential pressure transducers with a range of 0 kPa to 7 kPa (0 psi to 1,0 psi) are satisfactory. The transducer shall be capable of measuring the differential pressure to $\pm 0,1$ % of full scale.

7.6 Back-pressure regulators

The back-pressure regulator shall be capable of maintaining a pressure of 2,07 MPa to 3,45 MPa (300 psi to 500 psi). The stress applied to the cells shall take into account the back-pressure. For example, if the back-pressure is 3,45 MPa (500 psi), then the applied stress shall be 3,45 MPa (500 psi) greater to take into account the pressure exerted outward from the pistons.

7.7 Balance

The balance shall be capable of accommodating a minimum capacity of 100 g with a precision greater than 0,01 g.

7.8 Oxygen removal

The conductivity test fluid shall have the oxygen content reduced to simulate reservoir fluids and to minimize corrosion of test equipment. De-oxygenation can be accomplished with a two-reservoir system for the fluid. The first reservoir holds fluid for oxygen removal. This is connected to nitrogen gas that is bubbled through the fluid at low pressure below 103 kPa (15 psi) and at low rate. The nitrogen supply is first passed through an oxygen/moisture trap such as Agilent Model OT3-4²⁾ that has an efficiency to remove oxygen to less than 15 µg/l. An equivalent system can be made; this system allows nitrogen to pass through heated copper shavings at 370 °C (698 °F), where the copper reacts with the trace amounts of oxygen in the system forming copper oxide. An indicating trap, such as the oxygen trap by Chrom Tech, Inc. part # 10T-4-HP³⁾, after the oxygen-removal process allows for visual confirmation that oxygen has been removed. When the visual indicating trap is oxygen-saturated, both traps shall be replaced to maintain the efficiency of oxygen removal. The second reservoir holds the oxygen-free fluid; this is the supply reservoir for the pumping system.

All fluids in each reservoir are held in sealed, inert-gas pressurized containers to eliminate oxygen contamination from the air.

7.9 Temperature control

The test cell and proppant pack shall be maintained at the desired temperature ± 1 °C (± 3 °F). The temperature for the test conditions is measured in the temperature port of the conductivity cell (Figure C.1). This temperature is used to determine the fluid viscosity from Table C.1. The thermocouple assembly is split into a temperature-control device and a data-acquisition system or equivalent. The temperature control devices shall be programmable PID controllers and capable of self-tuning for different temperature conditions and flow rates.

A temperature of 121 °C (250 °F) is employed in the test for ceramics and resin-coated proppants and 66 °C (150 °F) for naturally occurring sands. The temperature for the silica-saturation vessel (see Annex B) should be 11 °C (20 °F) above testing temperature of 66 °C (150 °F) for naturally occurring sands. Sand 20 °C (35 °F) above 121 °C (250 °F) is used for resin-coated and ceramic proppants to ensure that the fluid is saturated with silica prior to reaching the cell. Care shall be taken to ensure that the fluid arriving to the cell is at the appropriate temperature. Tests using other fluids or temperatures can be of value in evaluating proppant pack conductivity.

2) Agilent Model OT3-4 is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 13503 and does not constitute an endorsement by ISO of this product.

3) Chrom Tech, Inc. part # 10T-4-HP is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 13503 and does not constitute an endorsement by ISO of this product.

7.10 Silica saturation and monitoring

It is critical to have a silica-saturated solution flowing through the proppant pack to prevent dissolution of the Ohio sandstone and proppant. To achieve this, a high-pressure cylinder with a minimum volume of 300 ml per 10 ml/min flow rate capacity, such as a Whitey sample cylinder 316L-HDF4⁴⁾, or equivalent equipped with 0,635 cm (0,25 in) female pipe ends is needed. For equipment setup, see Annex B.

8 Equipment calibration

8.1 Pressure indicators and flow rates

Pressure indicators in the test fluid-flow stream with back-pressure applied shall be calibrated initially and rechecked before each test. Constant-flow-rate pumps shall be tested at several flow rates with back-pressure applied with suitable flow meters, or accurate balance, containers and timing device (stop watch). High- and low-pressure transducers shall be zeroed before each run. Use only that portion of the transducer range that is repeatable and linear.

8.2 Zero pack width measurement

8.2.1 Purpose

To accurately measure the width of the proppant pack, the variations in sandstone thickness, the compressibility of sandstone and the compression and thermal expansion of the metal shall be taken into account.

8.2.2 Procedure

8.2.2.1 Using callipers, measure and record the thickness of the cores and metal shims. Mark the width of the core on the face of the core with a pencil. Two cores are placed in each cell. Match the cores so that the combined thickness of the ends of the cores is the same. Cores that measure more than 0,008 cm (0,003 in) from parallel shall not be used. If the bottom core is different from end to end, then the top core shall offset this difference, so the total core thickness at each end is identical.

8.2.2.2 A width adjustment factor or zero pack width shall be calculated at each closure stress and at temperature to be tested for each cell and for each lot of Ohio sandstone and square rings. Measure the vertical dimension of the complete test unit [$\pm 0,0025$ cm ($\pm 0,001$ in)] equipped with pistons, square rings, shims and sandstone cores, but without proppant, at each test closure stress level and temperature where the proppant will be tested. For each test, measure an initial zero width by measuring the vertical dimension of the pistons, shims and sandstone cores. This value is subtracted from the measured equipment and proppant values to obtain the actual width of the proppant pack.

8.2.2.2.3 Pistons for the baseline cell(s) shall be marked in the order in which they are stacked. Place the two matched sandstone cores in the cell and, if applicable, continue stacking the cells as in Figure C.1.

8.2.2.2.4 Heat the cells to the temperature at which the test will be run. Ramp closure stress at a rate of 689 kPa/min (100 psi/min).

8.2.2.2.5 Using telescoping gauges and digital callipers or equivalent, measure the piston from width slat to the bottom plate and from width slat to the top press plate or to the other width slat. All measurements shall be taken twice and both numbers shall be within $\pm 0,0050$ cm. Make another measurement 30 min after having made the first reading. Continue making measurements until the system reaches steady-state, e.g. the

4) Whitey sample cylinder of 316L-HDF4 is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 13503 and does not constitute an endorsement by ISO of this product.

measurements are within 1 % of each other. A minimum of three measurements shall be made. The last measurement shall be recorded. This procedure takes into account the compression of the sandstone cores and the expansion of metal at pressure and temperature. These values are used when calculating proppant pack widths (see Clause 12). Continue measurements at stress intervals indicated (see Clause 12) until maximum stress is reached.

8.3 Determination of cell width

Measure the inside of the cell at three places, two beside the high and low pressure ports and the third beside the middle port, using telescoping gauges and digital callipers. The three values are averaged. To determine the amount of proppant needed, multiply the average cell width by the amount of proppant desired divided by 38,1 mm. The following example is for a 9,76 kg/m² (2,00 lb/ft²) loading (see 10.2.4).

EXAMPLE (38,35 mm + 38,40 mm + 38,37 mm)/3 = 38,37 mm.

A two pound per square foot loading requires (63,00 g/38,10) × 38,37 = 63,44 g of proppant.

8.4 Hydraulic load frame

Calibration of the load cell shall be done at a minimum annually or when long term conductivity results are questionable. This type of device is preferred over use of hydraulic pressure gauges as a method of determining closure stress applied to the test cell. In some cases, a load cell is part of the system and must be calibrated by an external source.

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9 Leak tests

9.1 Hydraulic load frame

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The hydraulic system, i.e. lines, fittings and pumps, shall be tested initially and periodically thereafter to ensure there are no leaks. This may be accomplished by placing an appropriate block of high strength material having at least 64,5 cm² (10,0 in²) surface area between the platens at maximum load; shut in and observe to see if the pressure or load change is greater than ± 2 % of maximum reading during a 30 min period. If the pressure or load varies significantly, inspect all lines and fittings. If no line leaks are evident, there might be an internal leak in the control valve or the hydraulic ram.

9.2 Test fluid system

The initial complete test fluid system consisting of pump, lines, fittings and conductivity test unit shall be checked for leaks. To conduct a leak test, the conductivity test unit shall contain at least a monolayer of proppant material.

NOTE With no proppant between the platens, neither the square seal rings nor the downstream equipment can be tested.

Apply a closure stress of greater than 3,45 MPa (500 psi) to the conductivity unit and flow fluid through the system with a back-pressure 2,07 MPa to 3,45 MPa (300 psi to 500 psi). Close in the system and the pressure should not change more than 0,1 kPa (0,01 psi) in 5 min. Inspect all lines and fittings.

10 Procedure for loading the cells

10.1 Preparation of the test unit

10.1.1 Selecting the core

See 8.2.2